



# Particulate Materials Center

The Pennsylvania State University

## *Improving the processes underlying the production of powders and manufacturing with particulate materials*

### Center Mission and Rationale

Powder synthesis, dispersion, grinding, agglomeration, forming, and sintering are common unit operations in many particulate-based materials manufacturing industries such as protective coatings; composites; refractories; photographic; chemical processing; and electronic, magnetic, optical, and structural ceramics. Companies producing powders or manufacturing finished particulate materials share a common need for improved process consistency, efficiency, and reliability. The Particulate Materials Center was established to assist industry in achieving this goal by initiating research projects related to various stages of particulate materials manufacturing and processing, establishing educational programs for undergraduate and graduate students, and creating knowledge-transfer opportunities for industry. The research program seeks to improve particulate-based manufacturing by (1) developing new techniques for characterizing particle behavior at all levels of manufacture, (2) developing computational tools for efficient process simulation and evaluation, and (3) implementing these methods to obtain advanced process understanding and improved product manufacture. Information is transferred to our member companies through biannual meetings, visits, workshops, short courses, and future employees. The PMC faculty have appointments in the Colleges of Agricultural Engineering, Earth and Mineral Sciences, Engineering, Science, and Penn State's Interdisciplinary Materials Research Laboratory and are coordinated through Penn State's Intercollege Research Program.

### Research Program

The primary research programs are:

- **Particle Formation** — Particle formation processes offer the advantage of high volume production of powders with specific properties for applications such as advanced ceramics, electronic materials, refractories, intermetallic materials, chemicals, and others. The objective is to develop and improve the scientific and engineering understanding necessary for the design of powders synthesized by precipitation, emulsion, spray pyrolysis, hydrothermal, and sol-gel methods. Advances in this area require an integrated understanding of thermodynamics, chemistry, atomization dynamics, vapor phase reactions, chemical reactions, mixing and transport phenomena, coagulation and aggregation, and surface reaction processes. Manufacturing technologies under investigation include spray pyrolysis of unique composite particles, sol-gel synthesis of mixed metal oxides, low temperature techniques for  $<100^{\circ}\text{C}$  synthesis of biomaterials, precipitation of sub-100 nm particles, and spray drying of particle dispersions.
- **Particle Grinding and Classification** — This program seeks to establish model-based criteria and

engineering principles for the production of powders and slurries by fine grinding (typically  $<10\mu\text{m}$ ). The research program investigates the performance of grinding technologies such as attrition milling, media mills and autogenous grinding, and associated processes such as dispersion and classification. A goal is to establish comprehensive models for powder/slurry production systems. The impacts of interfacial phenomena on grinding device and classifier performance and on product powder/slurry properties are evaluated by particle size analysis and rheology. The ultimate goal is to provide engineering guidelines for equipment selection and process design along with operation and control parameters for the optimum production of fine powders and slurries in a variety of material systems. Fundamental studies on powder purity control during autogenous grinding, and submicrometer powder production by aggregated particle breakage are designed to develop these techniques as methods for low-cost production of submicrometer commercial powders.

- **Colloidal Dispersions** — The overall objective is to develop strategies for controlling the state of powder dispersion and aggregation. A particular interest is



**Professor Virendar M. Puri prepares for a test on a cubical triaxial tester, developed by Penn State's Powder Mechanics Lab to measure the stress state on powders in three dimensions.**

relating dispersant molecular structure to the colloidal properties, with the specific aim of identifying conditions yielding high solids-loaded slurries with minimum viscosity. With the decrease in particle size, surface characteristics and interfacial phenomena dominate fine particle dispersion. A particular interest is dispersion of  $<100\text{ nm}$  particles. The effects of dispersant molecular properties on particle dispersion are evaluated through adsorption studies and measurements of colloidal stability and rheology. The ultimate goal is to provide engineering guidelines for selection of reagent type and concentration for controlling the properties of

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dispersions, particularly in fine particle systems.

- **Powder Storage Flow and Handling** — The overarching goal is to develop and validate rational, principle-based engineering models for predicting the load-response of powders. Central to the model development is understanding the mechanics of dry, cohesive powders. Toward this end, time-independent and time-dependent constitutive equations for different bulk solids have been and are currently being investigated. Single- and multi-phase continuum theories have been developed and are being modified to accurately quantify powder behavior. Constitutive equation parameters have been measured using currently available testers as well as testers designed for accurate characterization of powders' stress-strain behavior. Engineering predictive models, based on the finite element method, have been developed for simulating the complex response of powders during storage and low pressure compaction. The finite element model (FEM) is being expanded to simulate incipient flow and dynamic flow behavior. The FEM will be an indispensable



**Professor Gary L. Messing (right) discusses spray pyrolysis, a technology for producing fine powder, with Dr. Roger Bagwell.**

principle-based design, analysis, evaluation, and synthesis tool for engineers and scientists in industry and academia.

- **Net Shape Forming** — This research program investigates the scientific and engineering aspects of novel suspension-based consolidation processes. The principal techniques under investigation are inorganic gel-casting and electrophoretic deposition. These approaches allow fabrication of advanced ceramics at reduced temperatures, with possibilities for unique composite architectures and film thickness variations unobtainable in other particle forming processes. Formation of ceramics by chemical reactions has broad applicability to forming monoliths and ceramic-matrix composites, and results in near net-shape without subsequent high temperature processing. For these reasons, unique ceramic-matrix composites can be

produced. Additionally, precursor phases can be interspersed with continuous fibers to produce high-toughness composites. Electrophoretic deposition (EPD) is a widely used, large-area industrial coating technique that permits great flexibility in fabrication of advanced materials through spatial control, connectivity, and functional gradients, with thickness control over an unlimited range of substrate shapes. EPD involves two basic processes: (1) electrophoretic migration of a charged particle and (2) the adhesion of particles to the electrode substrate. The control of these processes with manipulation of colloidal stability and the applied electric field strength permits the consolidation of particles on the substrate. One objective of this program is to develop an EPD process for continuous production of 1-10 mm thick films.

- **Press and Sinter Systems** — The objective of this program is to develop a fundamental understanding of all stages of ceramic press and sinter operations, from the selection and blending of powder, binder, plasticizers, and lubricants through the quick, accurate design of tooling and the evaluation of dimensional changes during sintering. To do this requires the development of test procedures for the measurement and characterization of granulated powders, the measurement of compact stress-strain behavior, and increased understanding of how powder characteristics and pressing additives affect behavior. The effect of these variables on the sintering and dimensional change of materials will also be taken into account. The computer model of the process incorporates test data to predict dimensional changes associated with all stages of ceramic press and sinter operations. One goal of the program is to establish better test procedures for each process step. Member companies will participate in activities that help establish the use of pressing software, measurement techniques, set-up and run parameters, expert systems, and non-destructive, ultrasonic evaluation of green parts. The ultimate goal is to develop tools that will reduce pressing defects as well as increase the manufacturing efficiency of powder pressing operations.
- **Particle Characterization Lab** — The Particle Characterization Lab has been established to assist our industrial partners, to support the research activities of the PMC, and to serve as a state-of-the-art characterization facility for routine and complex powder characterization problems, hands-on industrial short-course training, and undergraduate and graduate student education. Additionally, more specialized characterization needs and developments are provided through the individual laboratories of the PMC

faculty or other centralized facilities on campus. The Lab is partially supported by manufacturers of particle characterization equipment who are interested in supporting the PMC's research, education, and knowledge transfer programs.

#### **Special PMC Activities**

- **Education** — Industrial members' dues go directly to support M.S.- and Ph.D.-level research projects. The Center has also been successful in raising money to support an undergraduate research fellowship program. There are presently 11 students in this program working on research projects of interest to the industrial members, and we are working to expand this valuable activity. Additionally, the PMC has: established an industrial internship program for qualified graduate and undergraduate candidates; organized a university-wide seminar series designed to educate both undergraduate and graduate students, PMC faculty, and the university community at-large about the importance of powders and particulate materials in manufacturing; and is developing a seminar and separate lab credit courses for PMC-supported undergraduates and graduates as well as other students with an interest in particle processing technologies.
- **Knowledge Transfer** — The PMC regularly organizes short courses, seminars, and conferences in topical areas consistent with the research program and its members' interests. Existing program topics include Powder Production by Fine Grinding, Applied Powder Mechanics, Shaping of Technical Ceramics, Characterization of Powders and Consolidated Materials, and Engineering Challenges in Powder Metallurgy; other program topics are currently under development. These programs can be tailored for in-house use for interested companies. Apart from the Particle Characterization Lab, Penn State's extensive materials characterization capabilities are available.

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